EP 23668(2)

PCT WORLD INTELLECTUAL PROPERTY ORGANIZATION
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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 5 : A61F 13/02, B42D 15/00, B32B 31/00

A1

(11) International Publication Number: WO 93/07844

(43) International Publication Date: 29 April 1993 (29.04.93)

(21) International Application Number: PCT/US92/07569

(22) International Filing Date: 8 September 1992 (08.09.92)

(30) Priority data:
07/777,058 16 October 1991 (16.10.91) US

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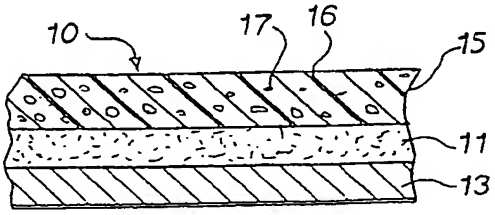
(81) Designated States: CA, JP, KR, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, SE).

Published
With international search report.

(54) Title: HIGH TEMPERATURE RESISTANT IDENTIFYING LABELS



(57) Abstract

An identifying label (10) which can be adhered at ambient temperatures to an object which is to be exposed to firing temperatures above 400 °C includes a base layer (11) of a bifunctional, inorganic pressure sensitive adhesive; a topcoat (15) receptive to identifying marking having a silicone binder (16); and, heat resistent, inorganic particles (17) in the topcoat (15) which interact with pyrolysis products formed by the firing to form an abrasion resistent identifying label (10). In a preferred embodiment, there is a woven cloth of fiberglass (14) between the base layer (11) and topcoat (15); and, a release liner (13) covering the underside of the base layer (11).

DOCID: <WO 9307844A1 I >

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SDOCID: <WO 9307841A1 >

HIGH TEMPERATURE RESISTANT IDENTIFYING LABELS

The present invention generally relates to labels. More particularly, it relates to identifying labels which can be applied to objects at ambient temperatures and which continue to serve as identifying labels after the objects and labels have been exposed to temperatures in excess of 400°C.

There is a need for heat resistant identifying labels that can be attached to objects, such as TV and CRT panels, which are fired by exposure to temperatures in excess of 400°C which would destroy ordinary labels. Depending upon their intended use, such labels also may have to possess other properties such as resistance to treatment acids and the like.

The Cornell U.S. Patent No. 3,669,787 discloses a sealing tape to be used in the production of cathode ray tubes. The tape comprises a layer of glass frit held together by a heat unstable organic binder that depolymerizes and volatilizes at temperatures below the melting point of the frit.

The Agarwal et al. U.S. Patent No. 4,693,920 discloses a label having a non-woven substrate, an adhesive coating on one side of the substrate and an organic resin coating on the other side of the substrate. The label is said to be capable of service up to 260°C.

DOCID: <WO 9307844A1 >

In the Yamano et al. U.S. Patent No. 4,971,858 a label is disclosed which includes an organic adhesive base; and an ink receiving top layer containing an inorganic material selected from metallic powder and metallic oxide powder, an organic binder, such as an acrylic resin, and glass frits. Similar labels, which in addition contain mica, are disclosed in European Patent Application 0 402 597 A1.

The tapes and labels disclosed in the aforementioned patents are useful for some purposes. However, the use of an organic binder that vaporizes or pyrolyzes can cause color control problems or pin holes or charring which can be troublesome for labels which are to be marked with bar codes or other identifying symbols. In addition, labels containing glass frits may not be resistant to treatment acids and can, if they contain lead, present disposal problems.

Therefore, there still is a need for an improved, heat resistant, identifying label.

It is the primary object of the present invention to disclose a novel identifying label which can be adhered to an object at ambient temperature and which will continue to function as an identifying label after exposure to temperatures in excess of 400°C.

The label of the present invention includes a base layer comprising a substantially inorganic, bifunctional pressure sensitive silicone adhesive which before, during, and after exposure to a firing temperature in excess of 400°C adheres the label to an object; an identifying marking receiving topcoat which comprises a silicone binder which before, during, and after exposure to the firing temperature binds to the base layer; and an effective amount of heat resistant, inorganic particles in the topcoat which coalesce with any pyrolysis products to provide an abrasion resistant, useful label. The label also may include a removable release liner which covers and protects the bottom of the pressure sensitive silicone adhesive base layer;

reinforcing materials to provide strength and pigments to provide contrast for the identifying markings.

In a preferred embodiment of the invention, the pressure sensitive adhesive of the base layer is a tacky silicone adhesive, the binder of the topcoat is a non-tacky silicone binder, the heat resistant, inorganic particles are titanium dioxide coated mica, and there is a woven fiberglass cloth reinforcing material between the base layer and the topcoat which provides the label with prefiring strength and support.

The manner in which the aforementioned and other objects of the invention are achieved by the practice of the present invention will be readily apparent to those skilled in the art from the description of the preferred embodiment and the drawings.

In the Drawings:

Fig. 1 is a top plan view of the preferred embodiment of a label of the present invention showing it before application to an object and firing;

Fig. 2 is a sectional view taken along line 2-2 in Fig. 1;

Fig. 3 is a view of the label after it has been applied to an object and fired;

Fig. 4 is an enlarged sectional view of a portion of the label and object of Fig. 3.

Fig. 5 is a view like Fig. 2 of another embodiment of the label of the present invention; and

Fig. 6 is a view like Fig. 2 of another embodiment of the label of the present invention.

In the preferred embodiment of the invention seen in Figs. 1 to 4, the label 10 comprises a base layer 11 of a bifunctional pressure sensitive silicone adhesive which before, during, and after firing at temperatures of about 400°C to above about 900°C binds the label 10 to an object 12 (seen only in Figs. 3 and 4). The underside of the base layer 11 is covered by a removable release liner 13, seen

only in Fig. 2, which is removed immediately prior to affixing the label 10 to the object 12.

Referring to Fig. 2, it can be seen that there is a woven cloth reinforcing material 14 overlying the top of the base layer 11 and an identifying-marking receptive topcoat 15 on top of the woven cloth 14.

As seen in Fig. 2, the topcoat 15 which comprises a non-tacky silicone containing binder 16 contains heat-resistant, inorganic particles 17 which do not melt or fuse upon exposure to firing temperatures. Similar particles 17 also are in the base layer 11. The purpose of the binder 16 before, during and after firing is to coat and fill any interstices in the woven cloth 14, and to bind the topcoat 15 to both the woven cloth 14 and the base layer 11. The purpose of the bifunctional adhesive in the base layer 11 before, during and after firing is to coat and fill any interstices in the woven cloth 14 and to bind the base layer 11 to the woven cloth 14, topcoat 15 and the object 12.

Referring to Figs. 3 and 4, it can be seen that after exposure to a firing temperature the pressure sensitive silicone adhesive of the base layer 11, the silicone binder 16 of the topcoat 15, and the inorganic particles 17 coalesce to form a ceramic 18 which is permanently bonded to the object 12.

In the embodiment of Fig. 5 there are heat resistant, inorganic particles 17 only in the binder 16 of the topcoat 15; and, in Fig. 6 an embodiment is shown in which there are both heat resistant, inorganic particles 17 and reinforcing fibers 19 in the topcoat 15.

The silicone pressure sensitive adhesive selected for the base layer 11 should have the following properties: First, the adhesive should be tacky enough to adhere the label to an object at ambient temperature. The preferred adhesive, is very tacky and when filled with 8.3% mica it has >30 oz/in. of adhesion to glass. The adhesive can be either cured with aminopropyltriethoxy silane, or benzoyl

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peroxide or used in the uncured state. If cured, the level of curing agent used should be chosen so that the adhesive retains the desired tack and adhesion properties. Second, the adhesive before, during, and after firing should adhere to both the woven cloth 14 and the topcoat 15. Third, the adhesive should interact with the heat resistant, inorganic particles 17 to form a permanent bond to the glass during the firing cycle. Fourth, the adhesive should not outgas significantly during the firing process. If the adhesive outgasses, decomposition products can either be trapped under the topcoat (causing bubbles in the label) or pass through the topcoat (causing pin holes in the label). Ideally there is only a small volume of gaseous pyrolysis products that slowly escape without causing any bubbles or pinholes to the label. The preferred adhesive meets these requirements.

The preferred silicone binder 16 for the topcoat 15 also should possess certain properties: First, the topcoat binder should be non-tacky at room temperature so that the label can be manufactured, imaged, and applied. The preferred binder is a silicone adhesive which is cross-linked to a tack-free, adhesion free surface by adding a stoichiometric excess of aminopropyltriethoxy silane. Second, the topcoat before, during, and after firing should adhere to both the adhesive layer 11 and the woven cloth 14. Third, the fired topcoat should be white or light colored. The preferred adhesive does not form any "charred" pyrolysis products so no discoloration occurs. Fourth, the fired topcoat should be hard and abrasion resistant. Fifth, the topcoat should allow for the escape of any pyrolysis products. The preferred silicone binder, the adhesive, satisfies these requirements.

Adhesives that can be used for the base layer 11 include without restriction:

1. The preferred adhesive which contains polytrimethylhydrosilyl-silicate.

2. An adhesive which contains dimethyl polysiloxane.
3. An adhesive which contains polydimethylsiloxane, hydroxy - terminated.

The adhesive which contains polytrimethylhydrosilyl-silicate has very high tack at room temperature. It can be used in an uncured state or it can be cross-linked with benzoyl peroxide, 2,4-dichlorobenzoyl peroxide or a metal octoate. The pressure sensitive adhesive which contains polydimethylsiloxane, hydroxy-terminated is similar in performance. The difference is that it is formulated to provide maximum adhesion to glass surfaces.

Compositions that can be used for the binder 16 of the topcoat 15 include without restriction:

1. The preferred adhesive which has been cross-linked with amino propyltriethoxy silane so that it has the desired low tack.
2. An adhesive which contains methylpolysiloxane. It is a silicone resin (non-pressure sensitive) which is a mixture of diphenyl, methyl, phenyl and phenyl methyl silicone resins. This resin is flexible at room temperature so the dried labels do not crack when flexed at room temperature. It can be filled with fillers and then drawn down (i.e., cast) onto a release liner. It forms a hard inextensible, smooth surface after firing at 450°C.
3. A silicone adhesive which contains a mixture of diphenyl, methyl, phenyl, and phenyl methyl resins. It is a benzoyl peroxide cured adhesive that has little tack at room temperature and becomes aggressively tacky at 200°F.

The heat resistant, inorganic particles are a necessary ingredient in the topcoat which after firing interact with the pyrolysis products of the pressure sensitive adhesive of the base layer 11 and the silicone

binder 16 to form a hard, abrasion resistant label. In the absence of an effective amount of at least about 2% by weight of the inorganic particles the label after firing lacks resistance to abrasion and structural strength. Preferrably, the topcoat composition will contain about 2% to about 33% by weight of the composition of inorganic particles ranging in size from about 1 μm to about 100)m in the topcoat. However, larger amounts might be useful for some purposes.

Representative of commercially available heat resistant, inorganic, particles 17 that can be employed are the following:

1. Zirconium Silica Hydrogel (65% SiO_2)
   5 μm Particle Size
2. Fused Silica
   5 μm Particle Size
3. Calcined Kaolin Clay
   (Anhydrous Aluminum Silicate)
   1.5 μm Particle Size
4. Calcined Aluminum Silicate
   1.3 μm Particle Size
5. Calcined Diatomaceous Silica
   8 μm Particle Size
6. Anhydrous Sodium Potassium Aluminum Silicate
   10 μm Particle Size
7. Fumed silica treated with dimethyl silicone fluid.
8. Pumice (72.0% SiO_2)
   20 μm Particle Size
9. Natural Zeolite
10. 99% Barium Sulfate
    1.9 μm Particle Size
11. Calcium Carbonate
    1.9 μm Particle Size
12. Magnesium Silicate (62.5% SiO_2)
    Low Micron Particle Size

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13. Amorphous Silica
3 μm Particle Size
14. Synthetic Sodium Aluminum Silicate
3 μm Particle Size
15. Mica Platelets Coated with Titanium Dioxide
4-75 μm Particle Size
16. Mica
10 μm Particle Size

The reinforcing material 14 and the reinforcing fibers 19 provide the label 11 with structural strength prior to firing so it can be removed from the release liner 13, and applied to an object without dimensional changes. In addition to being a woven cloth of fiberglass, the reinforcing material 14 may be a fabric of any other material which will provide the proper support for the other ingredients of the label prior to firing and which will after exposure to the firing temperatures not char, outgas, shrink or otherwise change so as to adversely affect the purpose and function of the label. The preferred reinforcing fibers 19 are also of fiberglass.

The preferred woven cloth is a fiberglass cloth which is preferred because of its minimal thickness of 1.1 mils. Minimal thickness is important because the label is to be imaged on its topcoat side with a barcode and corresponding alphanumeric information. For imaging, the surface of the label needs to be as smooth as possible. A thicker cloth produces a more distinct texture and image resolution is adversely effected. Also, this cloth needs to be heat cleaned. When glass cloth is produced the fibers are coated with an organic compound to make them less fragile and more processable into a cloth. This coating will volatilize at the 450°C firing temperature and could potentially cause bubbles or pinholes. Therefore, it is desirable, although not absolutely necessary, to pre-heat the cloth to about 400°C in order to pyrolyze any organics prior to using the cloth for label production.

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The release liner 13 can be any type of liner which has a coating that will release from the silicone adhesive or binder, such as a liner coated with a fluorinated silicone.

The preferred construction of the label is as follows:

Base Layer 11	=	100 Parts Adhesive
		10 Parts Mica
		20 Parts Toluene
Woven Cloth 14	=	Fiberglass
Topcoat 15	=	100 Parts Binder
		20 Parts Mica coated with TiO_2
		20 Parts Toluene
		6.6 Parts Catalyst
Release Liner 13	=	Liner coated with flourinated silicone

The label is preferably made by coating the base layer upon one side of a release liner coated on both sides with a release coat, laying a woven cloth upon the top of the wet base layer and then curing the woven cloth and the base layer to obtain a first product. Next, a topcoat comprising a silicone binder containing heat resistant, inorganic particles is coated upon the release coated top of a second release liner having an uncoated bottom to obtain a second product. The first and second products are then mated so that the topcoat of the second product contacts the cloth of the first product and the two products are laminated. The laminated product can be slit and die cut to form labels.

An alternative method of making the labels comprises coating a topcoat layer containing a silicone binder and heat resistant, inorganic particles upon the coated side of a release liner coated on one side with a release coat, laying a woven fiberglass cloth upon the top of the wet topcoat to obtain a first product. Next, a base layer comprising a pressure sensitive silicone adhesive is coated upon the release coated top of a second release liner coated on both sides to obtain a second product. The first and

second products are then mated so that the base layer of the second product contacts the fiberglass cloth of the first product and the two products are laminated. The laminated product can be slit and die cut to form labels.

The preferred identifying markings are imprinted upon a white topcoat 15 of the label 11 with a black ink, dye or toner which is permanent and which survives firing without adverse changes.

Especially preferred is an inked ribbon.

The identifying marking can be directly printed on the topcoat 15 by known printing techniques including screen printing, dot-matrix, ink jet, laser printing, laser marking, thermal transfer and the like. When the preferred topcoat binder 16 is used, it may be advantageous to powder or coat the topcoat 15 with $CaCO_3$ or a resin to optimize ink reception. It is preferred to print by thermal transfer the identifying markings using a ribbon that applies a black pigment to the white topcoat. In some instances, it may be advantageous to first print a bar code or other identifying marking on the topcoat and then to cover it with a protective material, such as a silicone resin, which after exposure to the firing remains or becomes transparent.

The invention is further illustrated by the following examples.

EXAMPLES 1-16

Labels were prepared having the following composition:

Base layer		
	Pressure Sensitive Adhesive	100 parts
	Particles	0 to 25%
	Toluene	10 parts
Reinforcing material		
	Woven Fiberglass cloth	
Topcoat		
	Binder (cured to low tack with aminopropyltriethoxy silane)	100 parts

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Particles - Mica coated with TiO_2	20 parts
Toluene	20 parts
Catalyst-(aminopropyltri-ethoxy silane)	6.6 parts

Release liner.

The base layer was cast down (2 mils) onto a two-sided release liner, the woven cloth was laid on top of the wet base layer to thoroughly saturate the cloth with adhesive, the resulting product was cured in an oven at 350°F for one minute and wound into a roll. A topcoat layer (2 mils) was cast down onto the coated side of a one-sided release liner, cured in an oven at 390°F for 3 minutes. The two products were brought together with the exposed topcoat of the second product and the woven cloth surfaces of the first product in contact, and hot laminated at 300°F. The one-sided release liner was then stripped and discarded and the product wound into a roll which was slit and die cut to form labels. The labels were printed by thermal transfer with identifying markings using an Iimak Hard 8 ribbon. A label was removed from the release liner and applied to a glass plate at room temperature. The glass plate and label were then fired at 450°C for 40 minutes. The appearance of the label and the bond of the label to glass plate were then evaluated.

The only differences between the compositions of the labels of the various examples was in the type and amounts of heat resistant, inorganic particles employed. In general, the best overall performance was achieved using particles which are some form of silica or a silicate.

Additional experiments were done. In these experiments the particles were added to the topcoat 15 and the base layer 11 contained 8.3% mica. The results obtained were comparable to those obtained except that the topcoat was not as white as when mica coated with titanium dioxide was used in the topcoat.

It will be apparent to those skilled in the art that a number of changes and modifications can be made without departing from the spirit and scope of the present invention. For example, in addition to the heat resistant particles, it may be advantageous for some applications to include glass frits or glass powder in either the base layer or topcoat. Therefore, it is intended that the invention be only limited by the claims.

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The embodiments of the invention in which an exclusive property or privilege is claimed are the following:

1. An identifying label which can be adhered at ambient temperature to an object which is to be subjected to firing at a temperature above about 400°C and which continues to function as an identifying label after such firing, said label comprising a base layer comprising a bifunctional, tacky, silicone pressure sensitive adhesive; and a topcoat receptive to identifying marking overlying the base layer, said topcoat comprising a silicone binder and inorganic, heat resistant, particles which do not melt or fuse as a result of the firing and which interact with pyrolysis products to form a stable label after firing.

2. A label of claim 1 which includes a removable release liner which protects the bottom of the pressure sensitive adhesive of the base layer.

3. A label of claim 1 which also contains a reinforcing material to provide prefiring strength.

4. A label of claim 3 which includes as a reinforcing material a woven cloth of fiberglass.

5. A label of claim 1 in which the particles are selected from inorganic silicates.

6. A label of claim 1 in which the particles are mica.

7. A label of claim 1 in which the particles are titanium dioxide coated mica.

8. A label of claim 1 which includes identifying markings on the topcoat.

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9. A label of claim 1 in which the silicone binder is a silicone adhesive which has been cured and is not tacky.

10. An identifying label comprising a base layer comprising a bifunctional, tacky, pressure sensitive silicone adhesive; a topcoat overlying the top of the base layer and comprising a silicone binder; heat resistant, inorganic particles in the topcoat; and, a release liner protecting the exposed bottom of the base layer.

11. An identifying label having a base layer comprising a bifunctional, tacky, silicone pressure sensitive adhesive containing heat resistant particles of mica; a topcoat comprising a silicone binder containing heat resistant, titanium dioxide coated, mica particles and overlying the top of the base layer; an intermediate reinforcing layer of woven fiberglass cloth at the interface between the top of the base layer and the topcoat; and, a release liner covering the underside of the base layer.

12. A method of preparing a high temperature resistant label, said method comprising coating a base layer of a pressure sensitive, silicone adhesive upon one side of a release liner coated on both sides with a release coat, laying a woven fiberglass cloth upon the top of the wet base layer, curing the thus formed cloth and base layer to obtain a first product; coating a topcoat comprising a silicone binder containing heat resistant, inorganic particles upon the release coated top of a second release liner having an uncoated bottom to obtain a second product; then mating the first and second products so that the topcoat of the second product contacts the fiberglass cloth of the first product; and laminating the two products to form a label.

13. A method of preparing a high temperature resistant label, said method comprising coating a topcoat layer containing a silicone binder and heat resistant, inorganic particles upon one side of a release liner coated on one side with a release coat, laying a woven fiberglass cloth upon the top of the wet topcoat to obtain a first product; coating a base layer comprising a pressure sensitive silicone adhesive upon the release coated top of a second release liner coated on both sides to obtain a second product; then mating the first and second products so that the base layer of the second product contacts the fiberglass cloth of the first product; and laminating the two products to form a label.

14. A method of preparing a high temperature resistant label, said method comprising coating a base layer of a pressure sensitive, silicone adhesive upon one side of a release liner coated on both sides with a release coat, curing the base layer to obtain a first product; coating a topcoat comprising a silicone binder containing heat resistant, inorganic particles upon the release coated top of a second release liner having an uncoated bottom to obtain a second product; then mating the first and second products so that the topcoat of the second product contacts the base layer of the first product; and laminating the two products to form a label.

15. A method of identifying an object which is to be subjected to firing which comprises attaching to said object a label of claim 1 before firing the object.

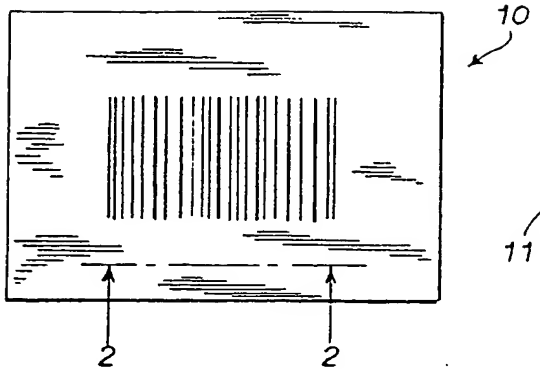

FIG. 1

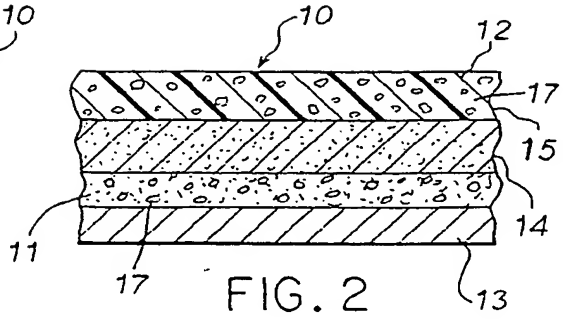

FIG. 2

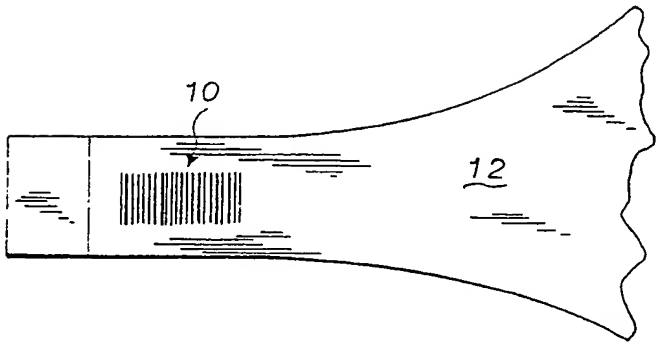

FIG. 3

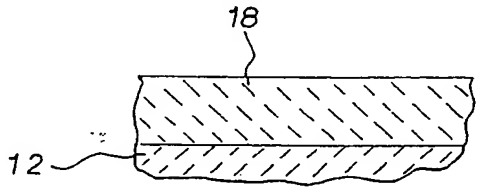

FIG. 4

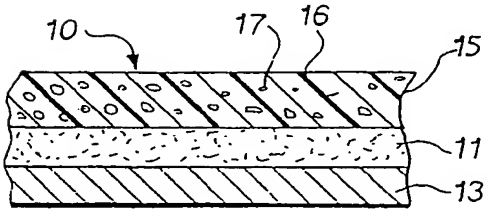

FIG. 5

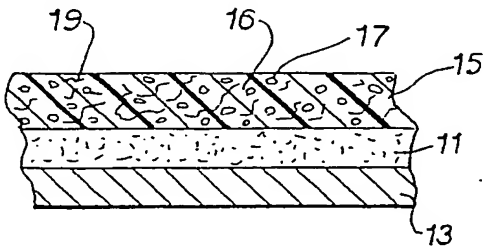

FIG 6

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/US92/07569

A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) :A61F 13/02; B42D 15/00; B32B 31/00
US CL :Please See Extra Sheet.
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 428/447, 40, 323, 324, 331, 354, 355, 915, 403, 404; 283/81, 101; 40/625, 630, 638; 156/280, 246, 249, 237, 241, 307.1

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	GB, A, 782,005 (MIDLAND SILICONES LIMITED) 28 AUGUST 1957, see page 1, lines 58-64 and page 2, lines 17-23.	1-15
Y	JP, A, 63-291962 (SHOWA ELEC WIRE KK) 29 NOVEMBER 1988, see English abstract.	1, 3-4
Y	US, A, 2,976,184 (BLATZ) 21 MARCH 1961, see column 2, lines 30-39.	2, 5-11, 15
Y	US, A, 4,693,920 (AGARWAL) 15 SEPTEMBER 1987, see column 3, lines 7-12.	2, 10-11
Y	WO, A, WO87/06252 (DOMNIKOV) 22 OCTOBER 1987, see page 10, line 34 through page 12, line 4.	12-14

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

*	Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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Date of the actual completion of the international search 21 OCTOBER 1992	Date of mailing of the international search report 04 NOV 1992
Name and mailing address of the ISA/ Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. NOT APPLICABLE	Authorized officer NASSER AHMAD Telephone No. (703) 308-2351

Form PCT/ISA/210 (second sheet)(July 1992)*

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A. CLASSIFICATION OF SUBJECT MATTER:
US CL :

428/447, 40, 323, 324, 331, 354, 355, 915, 403, 404; 283/81, 101; 40/625, 630, 638; 156/280, 246, 249, 237, 241, 307.1

Form PCT/ISA/210 (extra sheet)(July 1992)*

3DOCID: <WO___9307844A1_I_>